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(54) Title of the Invention: **Method for Producing Heat Resistant Dyed Cloth With Excellent Dye Fastness to Light**

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SPECIFICATION

1. Title of the Invention

Method for Producing Heat Resistant Dyed Cloth With Excellent Dye Fastness to Light

2. Claims

1. A method for producing a heat resistant dyed cloth, characterized by the carrier dyeing, at 120 to 100°C, of a cloth comprising mixed spun yarn of 80 to 50 wt% modified fully aromatic polyamide fibers and 20 to 50 wt% cellulose fibers.

2. A method for producing a heat resistant dyed cloth according to Claim 1, wherein the carrier comprises a phenolic carrier.

3. Detailed Description of the Invention

Field of Industrial Application

The present invention relates to a method for producing a dyed cloth with better dye fastness to light, heat resistance, flame retardant properties, and moisture absorption.

Fully aromatic polyamide fibers are generally difficult to dye because of the rigidity and crystallinity of the polymer chain. At present, a large amount of a carrier such as acetophenone must be used at a high dyeing temperature of at least 130°C in order to dye cloth comprising fully aromatic polyamide fibers. Despite the selection of such conditions, however, patterns sometimes do not permit a satisfactory color to be obtained. The selection of such conditions can also compromise the light fastness, moisture absorption, and hand. A conventional approach to address such drawbacks is the raw dyeing method, where a pigment is added to the polymer solution, which is particularly used for patterns which do not allow satisfactory dye concentrations to be obtained in cloth dyeing methods.

Raw dyeing is not used as commonly as cloth dyeing methods, however, because of the following drawbacks.

1) A larger number of patterns cannot be provided to meet market demand for greater pattern diversity. Operations are complicated by the need to switch starting materials and wash equipment in small-scale production of multiple lots, which results in higher manufacturing costs.

2) Manufacturing conditions in raw dyeing are not very flexible in cases where the pattern, color blend, and color preparation must be modified. Changing the polymerization conditions results in the need to readjust all processing conditions subsequent to polymerization, such as spinning, drawing, heat treatment, [illegible], knitting, weaving, and scouring.

The inventors perfected the present invention upon research, in view of the foregoing, on ways to produce heat resistant cloth that would have excellent dye fastness to light, moisture absorption, and hand, without any loss of the inherent performance of fully aromatic polyamide fibers, such as heat resistance and flame retardant properties.

That is, the present invention is a method for producing a heat resistant dyed cloth, characterized by the carrier dyeing, at 120 to 100°C, of a cloth comprising mixed spun yarn of 80 to 50 wt% modified fully aromatic polyamide fibers and 20 to 50 wt% cellulose fibers. Examples of fully aromatic polyamide fibers include those having a double layered structure comprising a readily dyeable skin layer and a less dyeable core layer (Japanese Unexamined Patent Application (Kokai) 55-142717), fully aromatic polyamide fibers comprising the copolymerization of xylylene diamine and a third component (Japanese Unexamined Patent Application (Kokai) 55-21406), fully aromatic polyamide fibers comprising the copolymerization of a third component such as toluylene diamine substituted with a methyl group as an alkyl substituent (Japanese Unexamined Patent Application (Kokai) 55-21046), and fully aromatic polyamide fibers comprising the copolymerization of a halogen-substituted diamine as the third component (Japanese Unexamined Patent Application (Kokai) 55-29516). Examples of cellulose fibers include typically well known fibers such as rayon fibers and cotton fibers, as well as modified fibers thereof. The cellulose fibers must be blended in a ratio of 20 to 50%. Less than 20% will not result in any improvement in the dyeing properties or the dye fastness to light. More than 50% will result in a woven material with lower heat resistance and

flame retardant properties after dyeing, and will not allow the inherent properties of the fully aromatic polyamide fibers to be fully exploited.

The fully aromatic polyamide fibers and cellulose fibers must be mixed spun to produce spun fibers. Parallel knitting or weaving will result in a loss of dye uniformity. Carrier dyeing is required. The use of a phenolic carrier is preferred as the carrier. Acetophenones can be used, but are undesirable because of problems with toxins in exhaust gas and wastewater. Paraphenyl phenols in particular have low toxicity and a high carrier capacity, allowing satisfactory dyeing to be achieved with low amounts of around 10%. The dyeing temperature must be no more than 120°C during the carrier dyeing. A temperature over 120°C will result in the deterioration of the cellulose fibers, with a marked loss of the hand of the woven material after dyeing. A dyeing temperature below 100°C will not afford satisfactory dyeing.

As should be evident by the preceding description, the present invention is intended to provide a method for producing a heat resistant dyed cloth having a high degree of dyeability and dye fastness to light, as well as a good hand, while preserving the inherent heat resistance and flame retardant properties of the aromatic polyamide fibers, as well as the moisture absorption of the cellulose fibers.

Specific examples of the present invention are given in the following embodiments. Parts and percentages in the examples are based on weight, unless otherwise specified. The heat resistant dyed cloth obtained by the method of the present invention was evaluated for dyeing properties, heat resistance, flame retardant properties, and dye fastness to light in the following manner.

1) Dyeing Properties

Dyeing properties were assessed by visually determining the depth of dyeing in judging whether the color was satisfactory or unsatisfactory for practical purposes.

2) Heat Resistance

The strength and elongation of the mixed spun fiber before and after treatment in the method of the present invention were measured using an Instron tension tester. A 30% or more decrease in strength was rated poor, while less than 30% was rated a pass.

3) Flame Retardance Properties

The burning time, smoldering time, and charred surface area with a 45° microburner (JIS L 1091A-1) were measured. A burning time of less than 3 seconds, smoldering time of less than 3 seconds, and charred surface area of less than 30 cm^2 were considered passing. All others were failed.

4) Dye Fastness To Light

This was assessed based on blue scale following 20 hours of exposure to a Fade-o-meter. Five ranks were used, where grade 5 indicates excellent, and grade 1 indicates poor. Grade 3 or higher was rated passing, while grade 2 and lower was failed.

Example 1

1525 parts isophthalic acid chloride were dissolved in 2500 parts tetrahydrofuran, and the mixture was cooled to 0°C. 102.3 parts metaxylylene diamine and 111.3 parts anhydrous sodium carbonate were dissolved in 2500 parts water, the mixture was cooled to 5°C, and the aforementioned tetrahydrofuran solution was added as the contents were vigorously stirred. After 3 minutes, 2500 parts water was added, the contents were stirred for another 5 minutes, and the resulting polymer was filtered off and washed 3 times with 2500 parts water, and was then dried at reduced pressure at 100°C. The resulting polymer had an I.V. of 0.73.

220 g of the resulting polymetaxylylene isophthalamide, and 124.7 g polymetaphenylene isophthalamide (I.V. = 1.80) polymerized from metaphenylene diamine and isophthalic acid chloride were dissolved in 1378.8 g *N*-methyl-2-pyrrolidone to prepare a spinning solution with a polymer concentration of 20.0 wt%. The spinning solution was then extruded at a rate of 4.0 m/mm from a nozzle (100 holes; hole diameter of 0.08 mm) and allowed to congeal in an inorganic salt solution based on calcium chloride, and the product was washed with water and then stretched 230-fold in boiling water, was then stretched 1.82-fold on 340°C hot plates, and was wound onto a winder.

The resulting aromatic polyamide fibers (2.5 d) were cut to a length of 51 mm, giving short fiber. 70 wt% of the short fiber and 30 wt% cotton were mixed spun to

produce two-ply yarn with a yarn count of 40, and the yarn was used as both warp and weft to weave a cloth with a density of warp 100/inch and weft 50/inch. The fabric was scoured under the usual conditions, preset, and then dyed under the following conditions using basic dye (C.I. Basic Blue 3).

C.I. Basic Blue 3	4% owf
$NaNO_3$	25 g/L
paraphenyl phenol	10% owf
pH	4
dyeing temperature	120°C
dyeing time	90 minutes

The cellulose fibers of the resulting dyed fabric were dyed under ordinary dyeing conditions, and were washed twice for 30 minutes while boiled in an aqueous solution containing 1 g/L nonionic surfactant. The resulting dyed fabric had a grade 4 dye fastness to light, as well as a good hand and good moisture absorption. The results are given in Table 1.

Comparative Example 1

Dyed fabric was obtained by carrier dyeing alone in the same manner as above except that 40 count yarn was produced by spinning 100% aromatic polyamide short fibers manufactured in the same manner as above. The results of performance evaluation are given in Table 1.

Example 2

Dyed fabric was obtained in the same manner as in Example 1 except that 40 count yarn was produced by mixed spinning of 50 wt% cotton and 50 wt% aromatic polyamide short fibers manufactured in the same manner as in Example 1. The results of performance evaluation are given in Table 1.

Comparative Example 2

Dyed fabric was obtained in the same manner as above except that 40 count yarn was produced by mixed spinning of 60 wt% cotton and 40 wt% aromatic polyamide short

fibers manufactured in the same manner as in Example 1. Although the dye fastness to light was excellent, the flame retardant properties were extremely poor. The results of performance evaluation are given in Table 1.

Comparative Example 3

Dyed fabric was obtained by spinning 100% aromatic polyamide short fibers to produce 40 count yarn, which was then dyed under ordinary dyeing conditions. The results of performance evaluation are given in Table 1.

Comparative Example 4

A plain weave (density of warp 100/inch, weft 50/inch) was produced from spun yarn (40 count two-ply yarn) obtained by mixed spinning of 30 wt% cotton and 70 wt% raw stock of polymetaphenylene isophthalamide fibers (by Konex Teijin).

A woven fabric dyed under the same conditions as in Example 1 had pale dyeing properties on the aromatic polyamide side and poor properties on the cotton side, with poor dye fastness to light. The results of performance evaluation are given in Table 1.

Comparative Example 5

A plain weave (density of warp 100/inch, weft 50/inch) was produced from spun yarn (40 count two-ply yarn) obtained by mixed spinning of 30 wt% cotton and 70 wt% aromatic polyamide produced in the same manner as in Example 1.

When the aromatic polyamide side was dyed using a carrier (paraphenyl phenol) under the same conditions as in Example 1, the aromatic polyamide side was pale, with the same poor dyeing properties with cotton as in Comparative Example 2. The dye fastness to light was also poor. The results of performance evaluation are given in Table 1.

Comparative Example 6

A plain weave produced in the same manner as in Example 1 was dyed at 140°C. There was a slight decline in the strength on the cotton side, with a considerable decrease in the fabric hand, rendering it unusable. The results of performance evaluation are given in Table 1.

Comparative Example 7

A plain weave obtained in the same manner as in Example 1 was dyed at 90°C. The results of performance evaluation are given in Table 1.

Table 1

	modified aramid	cotton	carrier (%)	dyeing temp. (°C)	light fastness (grade)	flame retardant properties	heat resistance	dyeing properties
Comp. Ex. 1	100	--	10	120	2 (poor)	good	good	good
Example 1	70	30	10	120	4 (good)	good	good	good
Example 2	50	50	10	120	4 (good)	good	good	good
Comp. Ex. 2	40	60	10	120	5 (good)	poor	poor	good
Comp. Ex. 3	0	100	10	120	5 (good)	poor	poor	good
Comp. Ex. 4	root aramid 70	30	10	120	2 (poor)	good	good	poor for root aramid
Comp. Ex. 5	modified aramid 70	30	0	120	2 (poor)	good	good	good
Comp. Ex. 6	70	30	0	140	4 (good)	good	poor for cotton	good
Comp. Ex. 7	70	30	0	90	2 (poor)	good	good	poor

(Note) modified aramid: modified wholly aromatic polyamide fibers; root aramid: unmodified wholly aromatic polyamide fiber